AD-A144 320 A METHOD FOR OBTAINING NEW DERIVED BENZIMIDAZOLES(U) 1/1
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W HAHN ET AL. 25 JUL 84 FTD-ID(RS)T-0794-84

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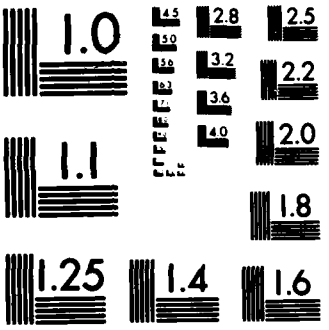

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Witold Hahn, Wieslaw Strzyzewski, Barbara Matecka

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EDITED TRANSLATION

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A METHOD FOR OBTAINING NEW DERIVED BENZIMIDAZOLES

by Witold Hahn, Wieslaw Strzyzewski, Barbara Matecka
authorized from the present patent: University of Lodz, Chemical Institute, Lodz (Poland)

The subject of the invention is a method for obtaining new derived benzimidazoles, 1,3 (benzimidazolylo-2) 1,3 dicyano-2-phenylopropene in example 1. This compound can find application in the creation of dyes.

According to the method, 2-cyanomethylobenzinimidazol is subjected to a condensation reaction with dibenzoyl. The reaction is performed in an alcohol medium in the presence of a base, under mild conditions at room temperature.

The product's mass spectrum shows a molecular ion corresponding to the molecular weight of 400. The summary sample, based on an elemental analysis, is comprised of the $C_{25}H_{16}N_6$ sample. This assignation proves that 2 molecules of 2-cyanomethylobenzimidazol and a half a molecule of dibenzoyl play a part in the formation of the product, which in sum is comprised of 419 mass units. From this it can be concluded that one atom of oxygen and three atoms of hydrogen must, which are comprised by 19 mass units, must be separated during the reaction. Precise studies of the reactive mixture has allowed the verification of the presence of benzaldehyde in it and the product of its condensation, described in the literature, with 2-cyanomethylo-benzimidazol in example 2. This data allows the course of the

reaction through the indirect creation of a-cyano-a(benzimidazolylo-2)-acetophenon in example 3 to be suggested. The infrared spectrum in the reaction's product in example 1 shows an unusual convergence with the infrared spectrum of the side compound expressed by example 2. This is understandable since the difference in the structure of the compounds in examples 1 and 2 depends on the substitution of the hydrogen atom in the compound in example 2 with 2-cyanomethylobenzimidazol, which is already present in this compound.

The infrared spectra are recorded in the Specord 71 IR apparatus, the NMR spectra--in the Tesla 80 MHz apparatus, the mass spectra--in the GCMF 2091 apparatus of the LKB firm of Switzerland.

The invention is illustrated by the following example, in which the percentage designates the weight percentage, and the temperature is given in Celsius.

Example. 3.3 g (around 3..015 mole of dibenzoyl dissolved in 110 cm^3 of exsiccated ethyl alcohol and .5 cm^3 of a 10% methanol solution of NaOH as a catalyst are added to 5 g (around .03 mole) of 2-cyanomethylobenzimidazol dissolved in 100 cm^3 of exsiccated ethyl alcohol. The mixture is left at room temperature for 48 hours. Then, the separated residue is filtered, washed several times with exsiccated ethynol and dried. 2.6 g of the product in example 1 is obtained with a melting point of 322-325°C. The reaction's yield amounted to 41.4%. The product after crystallization from the solution of N,N-dimethylophormamid-water possessed the form of yellow crystals with a melting point of 328-329.5°C.

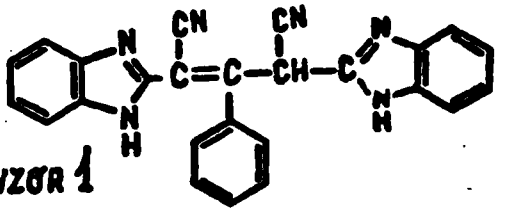
The elemental analysis:
for the $C_{25}H_{16}N_6$ sample (molecular weight 400)
--calculated: 75.06%C; 4.03%H; 21.01%N
--obtained: 74.70%C; 4.34%H; 21.27%N
IR spectrum (KBr): 3050-3430 cm^{-1} (NH); 2260 cm^{-1} (CN)
Mass spectrum: M/E/70 eV/; M^+: 400(100%)

Patent Specifications

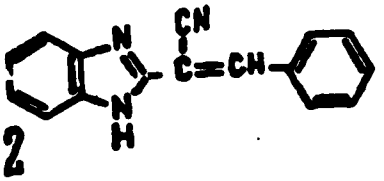
1. The method for obtaining new derived benzimidazol in example 1 is characterized by the fact that 2-cyanomethylobenzimidazol is subjected to a condensation reaction with dibenzoyl.

2. The method according to specification 1 is characterized by the fact that the reaction is performed in an alcohol medium in the presence of a base.

1. example



I WZÓR 1



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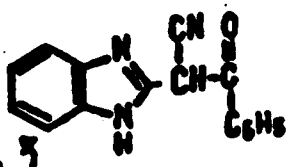
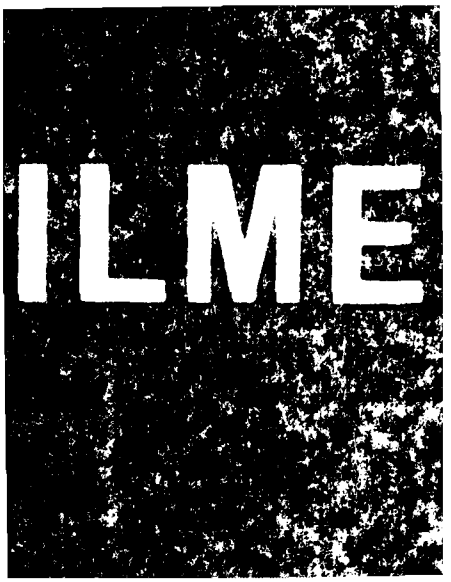


I WZÓR 3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